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THE PYROLYSIS OF POLYPHENOLIC PIGMENTS OF TOBACCO.

O. T. Chortyk, W. S. Schlotzhauer, R. L. Stedman, USDA, Eastern Utilization & Research Development Division, Philadelphia, Pennsylvania.

ABSTRACT

A pyrolytic study was made on certain high molecular weight polyphenolic tobacco pigments, which were isolated from Turkish tobacco and are known to contain rutin, chlorogenic acid, and amino acids. Pyrolysis of the dry pigment was conducted at 850°C., the approximate burn temperature of a cigarette. Most of the pyrolytic products were gases and volatiles not condensed in Dry Ice traps. The condensed products were fractionated into various groups, including a polynuclear aromatic hydrocarbon fraction. Using the usual isolation and identification procedures (thin-layer and paper chromatography, ultraviolet and fluorescence spectra), more than 12 polynuclears were found including anthracene, phenanthrene, fluoranthene, pyrene, chrysene, and benzo(a)pyrene as the major constituents. The yields of polynuclears obtained from the pigment were relatively high, e.g. about 1 mg. of benzo(a)pyrene from 1 g. of pigment. The use of a new gas solid chromatographic system for the separation of the higher polynuclear hydrocarbons in this study will be discussed.

REVIEW BY M. D. EDMONDS

During a conversation with Dr. Stedman concerning this paper, he stated that their pyrolysis work on polyphenolic pigments was in its preliminary stages.

The theory behind their pyrolysis is based on the formation of unstable free radicals which in turn undergo further reactions to form polynuclear hydrocarbons.

$$\text{Organic} \xrightarrow{\Delta} \text{R}\cdot \longrightarrow \text{Polynuclears}$$

Polyphenolic pigments were isolated from Turkish tobacco. The pigment fraction, consisting of rutin, chlorogenic acid, and certain amino acids, accounted for 4% by weight of the total sample.

The dried pigment was pyrolyzed at 850°C. The condensable products were collected and separated into acidic, basic, and neutral fractions. No work was reported for the acidic and basic fractions.

The neutral fraction was extracted with cyclohexane and separated into oxygenated neutrals, hydrocarbons, and other volatiles. The hydrocarbon fraction was separated into a fraction containing compounds of carbon and hydrogen and a fraction containing compounds of carbon, hydrogen and nitrogen. The latter fraction was not discussed.

The hydrocarbon fraction containing carbon and hydrogen was taken up in nitromethane and chromatographed on silica gel. The numerous fractions which were collected were chromatographed on paper and on thin layer plates (no mention of kind) and developed in methanol-ether-water or in toluene-ethanol-water systems. The following polynuclears were identified by R_f values and ultraviolet and fluorescence spectra:

benzene	pyrene*
naphthalene	1-methylpyrene
anthracene*	chrysene*
phenanthrene*	benzo(a)pyrene*
fluoranthene*	

* Major constituents

The quantitative data showed that the pigment produced BaP at a concentration of 1000 times that found in cigarette smoke.

A new solid gas chromatographic system for resolving higher molecular weight polynuclears was described. This was designated as "lithium chloride substrate," consisting of 20% lithium chloride on Chromosorb P.

A mixture of 28 polynuclears was resolved on the $LiCl_2$ substrate. The operating conditions were not given.

The advantages of this new substrate are: rapid resolution, quantitative data, decreased losses, and large-size sample analysis possible.